FCS02 – SOP for General Laboratory Procedures

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1. Scope

1.1. This method outlines the general procedures relating to the analysis of Controlled Dangerous Substances (CDS) in test materials. While these procedures provide general guidance and structure to the analytical process, due to the unpredictability of real-world samples, method variations may occur. In such cases, the deviations must be recorded as per Agency standards, either as a Minor or Major deviation (Defined in *DOM17 – Practices for Authorizing Deviations*).

2. Background

2.1. To establish the best practices for operations within the Forensic Chemistry Unit and to ensure conformance to the requirements of the Department of Forensic Sciences (DFS), the accreditation standards under ISO/IEC 17025:2017, and any supplemental standards.

3. Safety

3.1. The FCU follows *DOM13 – DFS Health and Safety Manual* and supplemental program guidelines.

3.2. Read Material Safety Data Sheets (SDS) to determine the safety hazards for chemicals and reagents used in the standard operating procedures.

3.2.1. Note: Do not add water to acid, only add acid to water.

3.3. Wear personal protective equipment (*e.g.*, lab coat, gloves, mask, eye protection), when carrying out standard operating procedures.

4. Materials Required

4.1. As required to perform analyses.

5. Standards and Controls

5.1. Reference materials shall, where possible, be traceable to the International System of Units (SI) units of measurement, or to certified reference materials (CRM). For seized drugs, this requirement is difficult to fulfill because the concept of traceability for drug standards is not internationally established and CRM's for drug analysis are not readily available or affordable.

5.1.1. Note: a certificate does not necessarily define a material as a CRM.

5.2. Standards are available from authorized vendors that manufacture ISO Guide 34 accredited products. The material shall be purchased from an ISO Guide 34 certified entity, whenever possible.

5.3. For quantitative determinations, different batches of reference material should be used for calibration and quality control. Where this is not practicable, the material can be sub-divided and each part assigned a specific purpose.

5.4. Assessment of reference materials

5.4.1. The FCU must ensure that each reference material is fit for purpose prior to use as a reference material.

5.4.1.1. Fit for purpose for qualitative work requires an assessment of chemical identity (structure, identifiable mass peaks, etc).

5.4.1.2. Fit for purpose for quantitative work requires an assessment of purity and its associated uncertainty of measurement.

5.4.1.3. The assessment shall be done on each lot of reference material.

5.4.1.4. The assessment and purpose of a reference material shall be documented. The documentation shall include the name of the individual who performed the assessment, the date of the assessment, verification test data, and details of all reference materials and reference data used.

5.4.1.5. Reference materials shall only be used for the purpose defined by the laboratory. For example, a reference material may be deemed suitable for qualitative but not quantitative determinations.

5.4.2. To be fit for purpose, the material must be assessed to indicate:

5.4.2.1. Pertinent analytical results, as applicable (i.e., chromatographic retention time, mass spectral results, infrared spectra).

5.4.2.2. Comparison of defining feature data to reference collection, published literature, or vendor-sourced information of standards.

5.4.3. These parameters may be described in a certificate, statement of analysis, data sheet, or label supplied with the material or may be determined by in-house analysis or reference to published literature.

5.4.4. For reference materials obtained from a provider accredited under ISO Guide 34, the information contained in the accompanying certificate is considered reliable and can be accepted as correct if the material is stored in accordance with the manufacturer's instructions.

5.4.5. For reference materials obtained from a provider not accredited under ISO Guide 34, the identity and purity information supplied by the provider shall be verified by analysis. Other information may be evaluated as needed.

5.4.5.1. Each new material shall be analyzed as per standard drug analysis to indicate criteria listed in 5.4.2.

5.4.5.2. The assessment may be completed prior to or alongside casework analysis as appropriate.

5.4.5.3. The laboratory shall assess the reliability of the information supplied with a reference material even if the material meets the definition of a certified reference material (CRM).

5.4.5.4. Examples of verification of chemical identity by analysis include:

5.4.5.4.1. Analysis and comparison of the results to peer-reviewed published data, data produced by a laboratory accredited under ISO/IEC

17025:2017, or to data produced from a previously verified reference material.

5.4.5.4.2. Evaluation of data from in-house structural elucidation analysis of the material.

5.4.5.5. Examples of verification of purity by analysis utilizing validated methods:

5.4.5.5.1. Comparison to previously verified material using Gas Chromatography-Flame Ionization Detection (GC-FID)

5.4.5.6. When verification by analysis is not possible, this shall be documented and the limitation expressed in the report, where applicable.

5.4.6. Where a reference material has no or limited supporting documentation or is produced in-house (by synthesis or from a case sample), then the chemical identity shall be determined in sufficient detail to demonstrate that it is fit for purpose. In addition, for quantitative work, the purity an associated uncertainty of measurement shall also be determined.

5.5. Expiration of Reference Materials

5.5.1. All reference materials shall have an expiration date.

5.5.2. If the material is not supplied with an expiration date by the manufacturer, an expiration date of 10 years from the date of receipt shall be assigned to the material.

5.5.3. If the expiration date passes before the material is fully used, then the material can be re-assessed, and the expiry date extended. The laboratory protocol for extending expiration dates shall be documented and should include analysis of the material.

5.5.4. The procedure for extending expiration dates includes the following steps:

5.5.4.1. A sample of the material with the expiration date to be extended shall be analyzed using GC-MS and/or GC-FID and compared to a non-expired material.

5.5.4.2. The expired material and non-expired material must meet GC-MS acceptance criteria as defined in *FCS09 - SOP for Operating and Maintaining GC-MS and GC-FID Instruments.*

5.5.4.3. The new expiration date for the reassessed standard shall be set at three months from the date of reassessment.

5.5.4.4. The results of this reassessment shall be released in a memo which shall be stored electronically along with the relevant data used for the reassessment.

5.5.4.5. Standards may be reassessed multiple times to further extend the expiration date.

5.5.4.6. A standard does not need to be expired before it undergoes a reassessment.

5.5.4.7. If expiry dates are not assigned to reference materials, the laboratory must have a documented protocol for assessing the validity of the reference material each time it is used.

6. Calibration

6.1. Calibration is only applicable to equipment or instruments performing quantitative measurements. Calibrations shall be performed as indicated per individual instrument or equipment SOP.

6.2. All other instrumentation and equipment used for qualitative purposes must be brought into good operating order as per individual instrument or equipment SOP.

7. Procedures

7.1. Analytical Procedures

7.1.1. The FCU shall have and follow documented analytical procedures.

7.1.2. The FCU shall have in place protocols for the sampling of evidence.

7.1.3. The FCU shall monitor the analytical processes using appropriate blanks, controls, or reference materials.

7.1.4. Method Validation

7.1.4.1. Method validation is required to demonstrate that methods are suitable for their intended purpose *(See DOM04 – Procedures for Validating Technical Procedures).*

7.1.4.2. Method validations will be varied based on the nature of procedures but shall generally follow guidelines as provided by

the Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) Recommendations.

7.1.5. The FCU shall have and follow documented guidelines for the acceptance and interpretation of data.

7.1.6. Acceptance criteria for a positive test result shall be defined in the corresponding method validations and operating protocols. In descending order of preference, acceptance criteria should be based on:

7.1.6.1. Comparison to data obtained from a suitable drug reference material analyzed under the same analytical conditions as the test/case sample. The reference material may be analyzed contemporaneously with test/case sample or as part of routine quality control.

7.1.6.2. Comparison to data obtained at a previous date (e.g., method validation, in-house library).

7.1.6.3. Comparisons to external reference data may be employed if a reference material is unavailable. External reference data shall be shown to be fit for purpose. The veracity of the data shall be considered and assessed. Factors to consider include:

7.1.6.3.1. Origin of the data

7.1.6.3.2. Validation of the data

7.1.6.3.3. Peer review of the data

7.1.6.3.4. Comparability of analytical conditions

7.1.6.4. The use of external reference data rather than a reference material should be documented and, where applicable, the limitation expressed within the report.

7.1.6.5. When neither reference materials nor external reference data are available, structural elucidation techniques may be employed providing the analyst has the appropriate skills for their interpretation. The absence of a reference material and external data shall be documented and the impact on the interpretation of reported results assessed.

7.1.7. When analysts determine the identity of a drug in a sample, they shall employ quality assurance measures to ensure the results correspond to the exhibit.

7.2. Chemicals and Regents

7.2.1. Chemicals and reagents used in drug testing shall be of appropriate grade for the tests performed.

7.2.2. Chemical and reagent containers should be dated and initialed when received and when first opened.

7.2.3. All chemicals and regents shall have an expiration date. If a material is not supplied with an expiration date by the manufacturer, an expiration date of 10 years from the date of receipt shall be assigned to the material.

7.2.4. Chemical and reagent containers shall be labeled as to their contents.

7.2.5. The efficacy of all test reagents shall be checked prior to their use in casework. Results of these tests shall be documented.

7.2.6. Some reagents, i.e., color test reagents, may be laboratory prepared. Refer to *FCS10 – SOP for Chemical Spot Tests* for specific procedures.

7.3. Instrumentation and Equipment Performance

7.3.1. Instrumentation

7.3.1.1. Instruments shall be routinely monitored to ensure that proper performance is maintained (i.e., by weekly maintenance and performance checks). This performance monitoring shall be completed and documented as defined by individual instrument SOPs (see below).

7.3.1.1.1. *FCS07 - SOP for Operating and Maintaining Analytical Balances*

7.3.1.1.2. *FCS09 – SOP for Operating and Maintaining GC-MS and GC-FID Instruments*

7.3.1.1.3. *FCS08 - SOP for Operating and Maintaining Nicolet iS50 Fourier Transform Infrared Spectroscopy (FT-IR) Instruments*

7.3.1.1.4. *FCS18 - SOP for Operating and Maintaining Spectrum Two Fourier Transform Infrared Spectroscopy (FT-IR) Instrument*

7.3.1.2. Monitoring shall include the use of reference materials, test mixtures, calibration standards, blanks, etc., as applicable.

7.3.1.3. Performance checks shall be performed on instruments that are moved from their normal operating positions, or instruments that have been previously taken out of service.

7.3.1.4. An annual preventive maintenance (i.e., once per calendar year) shall be performed to ensure instrument reliability and conformance to manufacturer's standards.

7.3.1.5. New instruments shall undergo a performance verification prior to being placed in service. Performance verifications shall ensure that results are produced as expected and defined by corresponding method validations.

7.3.1.6. The manufacturer's operation manual and other relevant documentation for instrumentation and equipment should be readily available.

7.3.1.7. If an instrument needs to be removed from the laboratory, *DOM13-Health and Safety* shall be followed. In specific, the Equipment Release Certification will be attached to the instrument after decontamination.

7.3.2. Equipment

7.3.2.1. Only suitable and properly operating equipment shall be employed. Equipment may be classified as critical or non-critical.

7.3.2.2. Critical equipment is defined as any type of equipment that may directly impact an analytical result in the event of a malfunction. These include, but are not limited to:

7.3.2.2.1. Hydrogen Generators and Water Purification System

7.3.2.2.2. Balances

7.3.2.2.3. Refrigeration units

7.3.2.2.4. Microscopes

7.3.2.2.5. All analytical instruments

7.3.2.3. Examples of non-critical equipment include:

7.3.2.3.1. Vortex

7.3.2.3.2. Hot plate

7.3.2.4. Performance parameters for critical equipment should be routinely monitored and documented.

7.3.2.5. Preventive maintenance shall be performed for critical equipment as per manufacturer's recommendations (or annually, i.e., once per calendar year) to ensure equipment reliability and conformance to manufacturer's standards.

7.3.2.6. The manufacturer's operation manual and other relevant documentation for each piece of critical equipment should be readily available.

8. Sampling

8.1. General Sampling Guidelines

8.1.1. This section addresses minimum recommendations for sampling of seized drugs for qualitative analysis. **NOTE:** For the purpose of this document the use of the term "statistical" refers to "probability-based."

8.1.2. The principal purpose of sampling is to answer relevant questions about a population by examination of a portion of the population.

8.1.3. By developing a sampling strategy and implementing appropriate sampling schemes, a laboratory will minimize the total number of required analytical determinations, while assuring that all relevant legal and scientific requirements are met.

8.1.4. One must be sure that what is sampled is truly representative of the total population. The analyst must take into consideration the homogeneity (or lack thereof) among drug packaging (bags, packets, capsules, etc.) and its contents. Careful visual inspections and personal experience are essential in determining the proper sampling procedure.

8.2. Types of Sampling Plans

8.2.1. Depending upon the inference to be drawn from the analysis for a multiple unit population, the sampling plan may be statistical or non-statistical.

8.2.2. Non-Statistical Sampling Plans

8.2.2.1. Non-statistical approaches are appropriate if no inference is to be made about the entire population.

8.2.2.2. Administrative Sampling Plan

8.2.2.2.1. One unit will be randomly selected and fully analyzed. Minimum recommendations for forensic drug identification shall be applied to the analyzed unit.

8.2.2.2.2. All remaining specimens will be left intact in case further analysis is required.

8.2.2.2.3. If further analysis is required and the analyzed unit is expended, an additional unit may be tested, as long as screening results are in agreement.

8.2.2.3. Percent-Based Sampling Plan

8.2.2.3.1. A percent-based approach to sampling may be employed, as directed by customer request. The specific percent to be sampled will be noted within the technical notes, e.g., worksheet, and the total number of units tested and total number of units not tested will be noted.

8.2.2.3.2. A percent-based system may be developed as part of a request, including determination of acceptance criteria for the unit determination. If no prior customer request is made for what defines a unique unit within a population, the chemist will decide based upon their experience.

8.2.2.3.3. As requested by the customer, either each item within the sampled population will be tested, or a composite will be made of the samples and a single analysis on that composite will be made.

8.2.3. Statistically Based Sampling Plans

8.2.3.1. A statistically based sampling plan (*e.g.,* hypergeometric distribution) will be used when inferences are made about the whole population. For example:

8.2.3.1.1. The probability that a given percentage of the population contains the drug of interest or is positive for a given characteristic.

8.2.3.1.2. The total net weight of the population is to be extrapolated from the weight of a sample.

8.2.3.2. The analyst may make statistical inferences about the entire population by analyzing a portion of multiple specimens.

8.2.3.3. Each unit comprising the sample shall be analyzed to meet the minimum recommendations for forensic drug identification, if statistical inferences are to be made about the whole population.

8.2.3.4. Inferences drawn from the application of the sampling plan and subsequent analyses shall be documented.

8.2.3.5. Hypergeometric Sampling Plan

8.2.3.5.1. Hypergeometric sampling is a statistically based model involving a defined confidence level with an associated probability of finding failures in a population. The hypergeometric model is used for specimens with no significant markings or labels (e.g., the contents of plastic bags and bag corners, vials, and glassine packets). This model may be used when the item requires a quantitative analysis.

8.2.3.5.2. Hypergeometric sampling may be used when additional analysis is requested.

8.2.3.5.3. The appropriate number of specimens within the population, as determined by Table 1, will be randomly selected to give a 95% level of confidence that at least 90% of the population contains the analyte in question.

8.2.3.5.4. Each specimen sampled will be analyzed separately and fully, unless otherwise directed by the customer.

Population (N) N_{max}=1000	**Proportion of Positives = 90% (Confidence Level=95%)**	**Population (N) N_{max}=1000**	**Proportion of Positives = 90% (Confidence Level=95%)**
1-10	ALL	34	18
11	9	35	18
12	9	36	19
13	10	37	19
14	11	38	20
15	12	39	20

16	12	40	18
17	13	41	18
18	14	42	18
19	15	43	19
20	12	44	19
21	13	45	20
22	14	46	21
23	14	47	21
24	15	48	21
25	16	49	22
26	16	50-59	23
27	17	60-69	23
28	18	70-79	24
29	18	80-89	25
30	15	90-99	25
31	16	100-199	27
32	17	200-1000	28
33	17		

Table 1. Hypergeometric Table for sampling of test items.

9. Calculations

9.1. See table 1 for hypergeometric sampling plan calculation table.

10. Uncertainty of Measurement

10.1. *FCS21 – Procedure for Uncertainty in Measurement*.

11. Limitations

11.1. Any limitations for analytical processes shall be clearly conveyed in specific method validations or SOPs.

11.2. Limitations must be clearly conveyed within the laboratory report.

12. Documentation

12.1. FCU Examination Worksheets

12.2. FCU Laboratory Report

13. References

13.1. This document is adapted from recommendations made by the Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) Recommendations (current

revision) for the use by the Department of Forensic Sciences (DFS) Forensic Chemistry Unit (FCU) in the District of Columbia.

13.2. OSAC Registry Standard: ASTM E2548-16 Standard Guide for Sampling Seized Drugs for Qualitative and Quantitative Analysis (Seized Drugs Subcommittee, April 2016).

13.3. Forensic Science Laboratory Quality Assurance Manual (Current Version)

13.4. DFS Departmental Operations Manuals (Current Versions)

13.5. FCU Standard Operating Procedures (Current Versions)